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34. (Added) The ball valve of claim 28 wherein the coating has a ground and polished finish.
35. (Added) The ball valve of claim 34 wherein the coating is deposited by thermal spray application of a powder comprising spherical agglomerates in a size range from 10 to 45 microns comprising a mixture of ultrafine particles of less than 300 nm.
36. (Added) The process of claim 20, wherein the solution is at least 98 percent sulfuric acid.

### REMARKS

The Office Action dated April 10, 2003, required restriction under 35 U.S.C. § 121 to one of the following: Group I, Claims 1-2, and 22; Group II, Claims 3-8 and 23; Group III, Claims 9-11 and 24; Group IV, Claims 12-20, 25 and 26; or Group V, Claims 21 and 27.

Pursuant to Applicant's provisional election of Group IV, the Examiner rejected claims 12-20 under 35 U.S.C. § 112, first paragraph.

### Confirmation of Election

Applicant maintains the election of Group IV as the invention to be prosecuted.

### Objections

The examiner has objected to paragraph [0022] of the specification as being unclear. The examples given,  $\text{TiO}_2\text{-}20\text{Ta}_2\text{O}_5$  and  $\text{TiO}_2\text{-}45\text{ZrO}_2$ , are illustrative of the type of chemical compositions that can be employed to coat the titanium ball valves. The specification states that the relative quantities of the second phase can be between 5 and 45 vol%. The first example is made up of 80 vol%  $\text{TiO}_2$  mixed with 20 vol%  $\text{Ta}_2\text{O}_5$  and the second example is 55 vol%  $\text{TiO}_2$  mixed with 45 vol%  $\text{ZrO}_2$ . The artisan of ordinary skill in this art readily appreciates that the number preceeding the second compound formula represents the respective volume percentage of that compound that is present. The objection should be withdrawn.

Applicant appreciates the examiner's noting of the typographical errors in paragraphs [0028], [0034], [0037], and [0038] in the specification. These errors seem to have arisen from the removal of hyphens from the printed text during electronic filing. The typographical errors have been corrected in the above amendments. No new matter has been added.

The office action objects to paragraph [0042] in the specification, requesting an explanation for the term  $HV_{0.3}$ . The skilled artisan understands that HV stands for "Hardness Vickers," signifying that the Vickers test is employed to determine the microhardness of the material. The Vickers test employs a square base diamond shaped stylus. The Vickers test is distinguished from the other commonly employed test, the Knoop test, which employs a rhombus shaped stylus. The subscript 0.3 refers to the load on the stylus in kilograms. Here, a 300g load was placed on the stylus when measuring the hardness of the material. Based upon the indentation made and the load placed on the stylus, the microhardness value is determined. A description of the test and an example of the use of HV obtained from the INSTRON website (located at [http://www.instron.com/applications/test\\_types/hardness/vickers.asp](http://www.instron.com/applications/test_types/hardness/vickers.asp)) has been included with this response.

The office action objects to the specification as failing to provide clear and exact terms as to enable any person skilled in the art to make and/or use of the claimed invention. Specifically, the examiner believes there are discrepancies regarding the use of "ultrafine" and the dimensions used to define ultrafine particles found in paragraph [0007], at page 5 (line 10), and claim 17. At paragraph [0007], the term "ultrafine" is used to define materials having physical features less than 300 nm. At paragraph [0011] the specification details the materials used directly in the spray process as being spherical agglomerates ranging in size from 5 to 100 microns, but preferably between 10 and 45 microns, prepared from a mixture of ultrafine titania particles of less than 300 nm. At paragraph [0014] (line 10), the specification states that the preferable grain size of the coating is less than 500 nm. This does not state that the grain size is considered to be "ultrafine." This is merely illustrative of one aspect of the present invention. Furthermore, in paragraph [0012] the applicant states that another aspect of the invention is "ultrafine" and "preferably nanostructured" titania coating. The grain growth is a function of the

material and a function of the size of the particles used to prepare the coating. The references to the particle size as being "ultrafine" and to one aspect of the invention being a coating of preferably less than 500 nm found in paragraphs [0007], [0012], and [0014] are distinct of one another. This objection is likewise inappropriate and should be withdrawn.

#### § 112 Rejections

Claims 12-20 stand rejected under 35 U.S.C. §112, as lacking enablement. The Examiner states that the rejection is made on the same basis as the objection discussed above regarding the use of the term "ultrafine." The Applicant has considered the rejection in view of the Examiner's comments and respectfully disagree that the claims, as amended, lack enablement, for the same reasons noted above.

The examiner objects to the specification and rejects claims 12-20 as failing to provide clear and exact terms as to enable any person skilled in the art to make and/or use of the claimed invention. Specifically, the examiner believes there are discrepancies regarding the use of "ultrafine" and the dimensions used to define ultrafine particles found in paragraph [0007], at page 5 (line 10), and claim 17.

At paragraph [0007], the term "ultrafine" is used to define materials having physical features less than 300 nm. This is a term chosen by the Applicant and is defined as stated above. The term "nanostructured" is defined as materials having physical features less than 100 nm. At paragraph [0011] the specification details the materials used directly in the spray process as being spherical agglomerates ranging in size from 5 to 100 microns, but preferably between 10 and 45 microns, prepared from a mixture of ultrafine titania particles of less than 300 nm. At paragraph [0014] (line 10), the specification states that the preferable grain size of the coating is less than 500 nm.

The statement in the specification that the preferable grain size of the coating is less than 500 nm is not inconsistent with the definition of ultrafine. This is just one aspect of the current invention and it is not limited to ultrafine grain size. Grain size is determined by a variety of factors and conditions, including the particle size of both the TiO<sub>2</sub> and any secondary immiscible phase, as well as the size of the agglomerate used in the spraying process. Depending on processing conditions that affect grain growth, it

is possible to produce coatings that have grain size greater than 300 nm even when using ultrafine particles as starting material. While one aspect of the invention is directed to titania coatings having nanostructured and ultrafine grain sizes, it is also directed to titania coatings having grain size of less than 500 nm.

In claim 12, the term ultrafine has been removed and is incorporated into dependent claim [28] to remove any inconsistencies that may be present.

In view of the arguments made regarding the specification, the Applicant respectfully submits that the claims do not lack enablement and respectfully requests that the rejection under 35 U.S.C. §112, first paragraph be withdrawn.

Support for the amendment of claim 20 is found in paragraph [0015] of the specification, inter alia. No new matter is presented.

The Applicant respectfully notes that the Examiner failed to initial the Electronic IDS submitted previously. Applicant submits herewith a copy of the Electronic IDS and respectfully requests that the Examiner consider the references contained therein.

The Commissioner is authorized to charge any fees associated with this communication to deposit account 501285. If the Examiner has any questions or comments regarding this communication, the undersigned can be contacted to expedite the resolution of this application. Further examination of the application and reconsideration of the claims as amended and the allowance thereof are respectfully requested.

Respectfully submitted,



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**Marked-Up Version Of Specification Showing Changes Made**

[0014] Yet another aspect of the invention is the provision of a ball valve for handling very corrosive fluids and abrasive solid particles in a pressure leaching process. The ball valve includes a valve body, a ball centrally positioned in the valve body and having a central passage rotatable in the valve body between open and closed positions, and at least one seat disposed between the ball and the valve body. The ball and seat each comprise a titanium substrate and an ultrafine, preferably nanostructured titania coating. The coating can have a titania phase and a phase immiscible with the titania phase in a proportion effective to inhibit grain growth. The immiscible phase preferably comprises from 5 to 45 percent by volume of the coating. The immiscible phase can be selected from zirconia, tantalum oxide, boron carbide, silicon carbide, titanium carbide, diamond and combinations thereof. The coating can have a ground and polished surface. The coating can have a thickness from 100 to 500 microns, or preferably when it has a ground and polished surface, a thickness of from 100 to 200 microns. The titania coating preferably has a grain size less than 500 nm. The coating is preferably deposited by thermal spray application of a powder comprising spherical agglomerates in a size range of from 10 to 45 microns comprising a mixture of ultrafine particles of less than 0.3 microns.

[0028] In a preferred embodiment, the thermal spray process comprises the atmospheric plasma spray (APS) process. In the APS process, a jet of gas is heated by an electric arc to form a plasma jet. Powder feedstock is injected into the plasma jet to heat the particles and to accelerate them towards a substrate to form a coating. The spray parameters preferably include a gun current of 400 to 500 amps, a primary gas (argon or nitrogen) flow rate of 36 to 48 SLPM, a secondary (hydrogen) gas flow rate of 7 to 12 SLPM, a spray distance of 50 to 80 mm, a powder feed rate of 36 to 60 g/min, a maximum substrate surface temperature of 200°C, and a spray thickness of 125 to 500 microns. The coated substrate is then allowed to cool to ambient temperature.

[0034] Example 1 A nanostructured titania on titanium ball valve was made by coating the Grade 5 titanium seats **112**, **114** and ball **108** of the valve shown in

Figs. 2-5. An atmospheric plasma spray (APS) gun was used, manufactured by Sulzer Metco, model number 7M with a Sulzer Metco feeder, model number 9MP. Prior to applying the coating, the component surface was grit blasted using alumina (20 to 36 microns) to 2-3 mils and heated to above 100 °C. The powder used was ultrafine titania agglomerates that had been prepared according to specifications (agglomerates approximately 5 to 45 microns, ultrafine particles approximately 300 nm) by material suppliers. The powder was applied by repeatedly passing the flame over the parts, allowing the parts to cool slightly between passes. The gun current was 400 to 500 A, the primary gas (argon or nitrogen) flow rate was 36 to 48 SLPM, and the secondary gas (hydrogen) flow rate was 7 to 12 SLPM. The powder injection feed rate was 36 to 60 g/min, and the spraying distance was 50 to 80 mm. The part surface temperature was maintained below 200 °C throughout the spray process. The coated ball valve parts were ground and polished to 8 RMS.

[0037] Example 4 An agglomerated ultrafine composite powder for thermal spray application was produced by: 1) milling mixtures of commercial (micron size range)  $\text{TiO}_2$  and 20 volume percent  $\text{Ta}_2\text{O}_5$  powders down to below 300 nm particle size range; and 2) spray drying with appropriate (1 to 6 weight percent of total solution) organic binders to form spherical agglomerate powder. The milling was carried out in an aqueous-based liquid medium with 30 to 35 weight percent solids. Organic binders used in spray drying included polyvinyl alcohol (PVA) or carboxymethyl cellulose (CMC). The spray-dried powder consisted essentially of spherical agglomerates, in the size range of 10 to 30  $\mu\text{m}$ .

[0038] Example 5 An agglomerated ultrafine composite powder for thermal spray application was produced by milling mixtures of commercial (micron size range)  $\text{TiO}_2$  and 45 volume percent  $\text{ZrO}_2$  powders down to below 300 nm particle size range and spray drying with appropriate (1 to 6 weight percent of total solution) organic binders to form spherical agglomerate powder. The milling was carried out in an aqueous-based liquid medium with 30 to 35 wt% solids. Organic binders used in spray drying included polyvinyl alcohol (PVA) or carboxymethyl cellulose (CMC). The spray dried powder consisted essentially of spherical agglomerates, in the size range of 5 to 45  $\mu\text{m}$ .

**Marked-Up Version Of Claims Showing Changes Made**

1. (Canceled)
2. (Canceled)
3. (Canceled)
4. (Canceled)
5. (Canceled)
6. (Canceled)
7. (Canceled)
8. (Canceled)
9. (Canceled)
10. (Canceled)
11. (Canceled)
12. (Amended) A ball valve for handling very corrosive fluids and abrasive solid particles in a pressure leaching process, comprising:
  - a valve body;
  - a ball centrally positioned in the valve body and having a central passage rotatable in the valve body between open and closed positions;
  - at least one seat disposed between the ball and the valve body;
  - wherein the ball and seat each comprise a titanium substrate and a [ultrafine] titania coating.

13. The ball valve of claim 12 wherein the coating comprises a titania phase and a phase immiscible with the titania phase in a proportion effective to inhibit grain growth.
14. The ball valve of claim 13 wherein the immiscible phase comprises from 5 to 45 percent by volume of the coating.
15. The ball valve of claim 13 wherein the immiscible phase is selected from zirconia, tantalum oxide, boron carbide, silicon carbide, titanium carbide, diamond and combinations thereof.
16. The ball valve of claim 12 wherein the coating has a thickness from 100 to 500 microns.
17. The ball valve of claim 12 wherein the titania has a grain size less than 500 nm.
18. The ball valve of claim 12 wherein the coating has a ground and polished surface.
19. The ball valve of claim 18 wherein the coating is deposited by thermal spray application of a powder comprising spherical agglomerates in a size range of from 10 to 45 microns comprising a mixture of ultrafine particles of less than 0.3 microns.
20. (Amended) A pressure acid leaching process comprising alternately opening and closing the ball valve of claim [11]12 to respectively allow and stop passage of an acid leach mixture comprising abrasive particles in a solution of [at least 98 percent] sulfuric acid at a temperature above 250°C and pressure above 4000 kPa.



21. (Canceled)
22. (Canceled)
23. (Canceled)
24. (Canceled)
25. The invention of claim 12 wherein the ultrafine particles are nanostructured.
26. The invention of claim 20 wherein the ultrafine particles are nanostructured.
27. (Canceled)
28. (Added) The ball valve of claim 12 wherein the coating has a grain size less than 300 nm.
29. (Added) The ball valve of claim 12 wherein the coating has a grain size less than 100 nm.
30. (Added) The ball valve of claim 28 wherein the coating comprises a titania phase and a phase immiscible with the titania phase in a proportion effective to inhibit grain growth.
31. (Added) The ball valve of claim 30 wherein the immiscible phase comprises from 5 to 45 percent by volume of the coating.
32. (Added) The ball valve of claim 30 wherein the immiscible phase is selected from zirconia, tantalum oxide, boron carbide, silicon carbide, titanium carbide, diamond and combinations thereof.

33. (Added) The ball valve of claim 28 wherein the coating has a thickness from 100 to 500 microns.
34. (Added) The ball valve of claim 28 wherein the coating has a ground and polished finish.
35. (Added) The ball valve of claim 34 wherein the coating is deposited by thermal spray application of a powder comprising spherical agglomerates in a size range from 10 to 45 microns comprising a mixture of ultrafine particles of less than 300 nm.
36. (Added) The process of claim 20, wherein the solution is at least 98 percent sulfuric acid.